PROJECT NUMBER: 6502
PROJECT TITLE : Process Monitoring and Real Time Measurements
PROJECT LEADER : R. W. Kanipe
PERIOD COVERED : July, 1991

PROCESS METHODS DEVELOPMENT

A. **Objective:** To develop a QA procedure for acceptance of adhesive shipments at Cabarrus.

B. **Results:**

Various levels of methanol, formaldehyde, acetic acid, and ammonia were added to selected adhesive samples to evaluate the effects on gas-phase and/or liquid-phase FTIR spectra as compared to reference spectra. These compounds have been identified as typical contaminants in adhesives. Results showed that 1% methanol and 2% acetic acid could be visually detected in the liquid-phase (ATR FTIR) spectra. For the gas-phase spectra, methanol was visually detected at 0.02% for an adhesive without vinyl acetate and 0.1% for an adhesive with vinyl acetate. Acetic acid was visually detected at 8% and 1% for adhesives with and without vinyl acetate, respectively. Ammonia was visually detected at ~0.2% in the vinyl acetate adhesive sample. Results for samples spiked with formaldehyde were inconclusive.

Experiments were completed to determine the stability of the twelve reference adhesives during the development stages of the headspace FTIR method. The samples had aged two months since the initial stages of development. Headspace for three of the adhesive samples did show reduced FTIR absorbance spectra when compared to reference spectra. For two of the samples, the reduction was primarily attributed to excessive sample handling during the two month period. Spectra for the remaining nine adhesives showed no significant differences when compared to reference spectra.

C. **Conclusions:** In general, typical contaminants in adhesives generate spectral differences which are visually detectable at certain levels when compared to reference spectra. Sample integrity was acceptable during the development stages of the headspace method; however, indications are that the adhesives do not have an infinite shelf life and sample integrity may be affected by excessive sample handling.

D. **Plans:** Determine the acceptable level of spectral variation between sample and calibration data. Continue to develop software to facilitate routine sample analysis.

E. **References:**

1. Parrish, M., PM Notebook 9106, pp. 7-12.

2. Lewis, W., Lewis, PM Notebook 9095, pp. 23-28.